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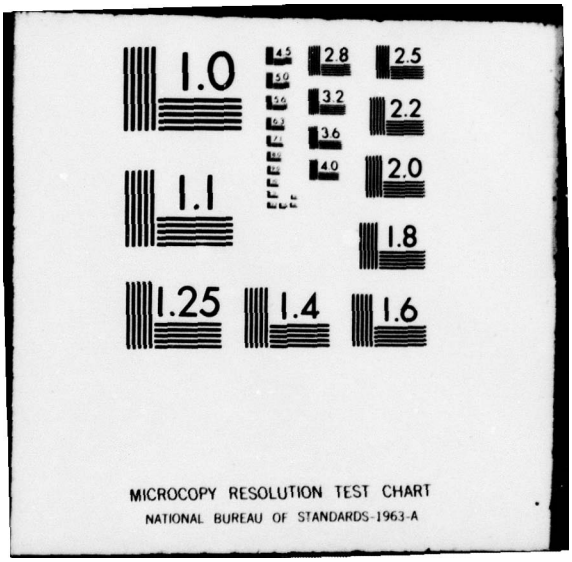
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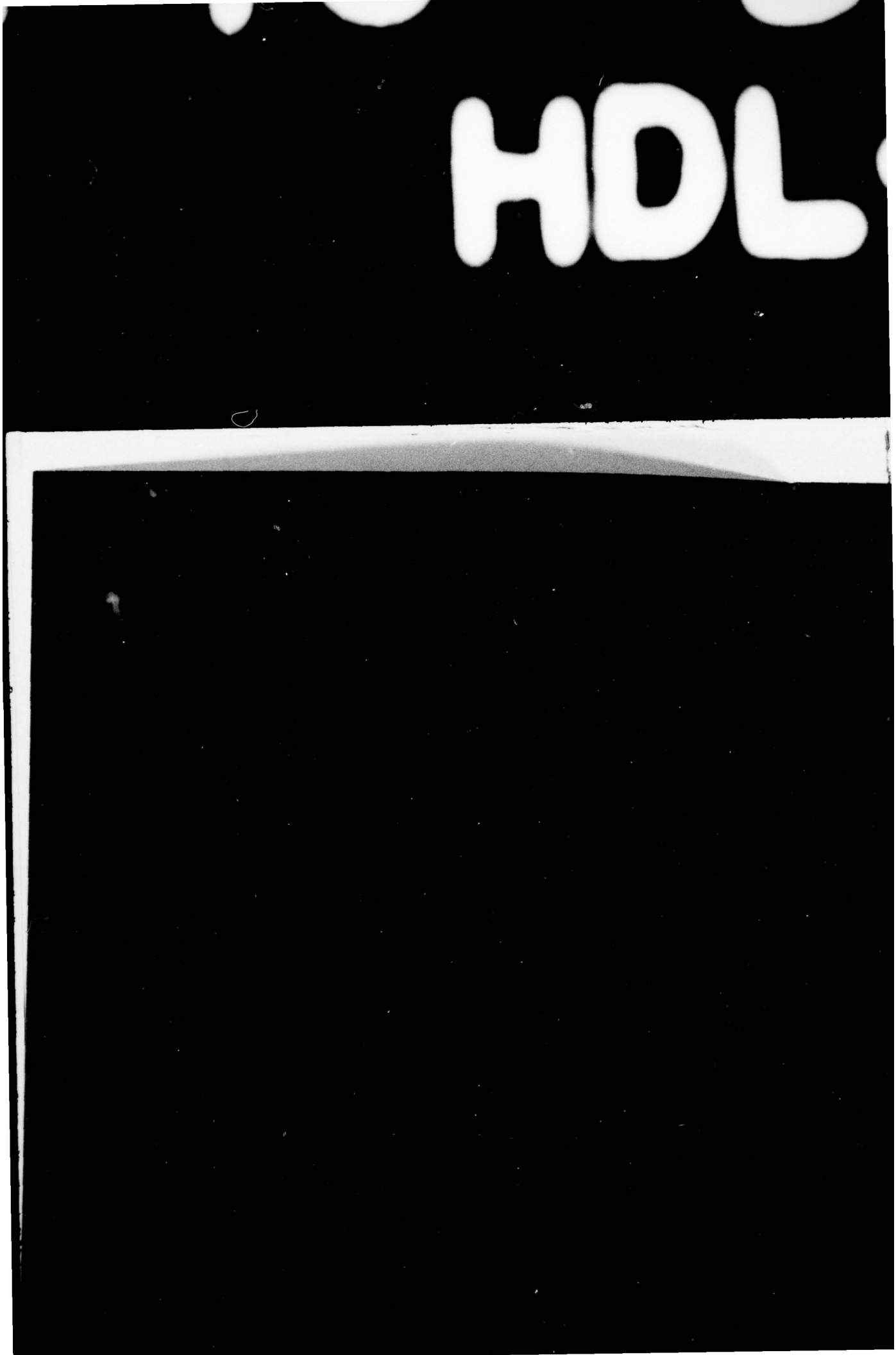
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Two antistatic coating formulations have been developed at the Harry Diamond Laboratories for the plastic nose cones of artillery fuzes. Both are conductive carbon-filled epoxy compounds. Formulation HDL900-3, developed in 1971, uses 2-ethyl-4-methylimidazole (EMI-24) as the curing agent. Formulation HDL900-6, developed as an alternative, uses m-phenylenediamine as the curing agent. Thermal stability, resistivity, and durability

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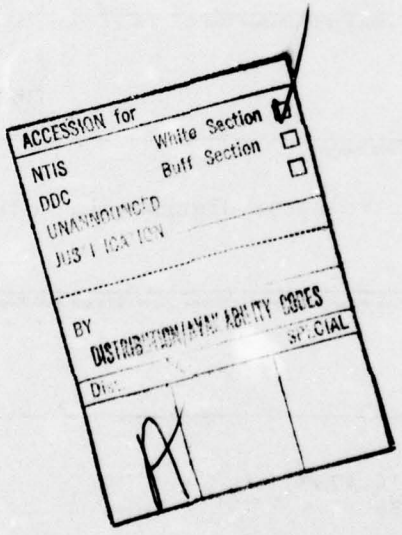
20. Abstract (Cont'd)

are the main criteria used to evaluate the formulations. The EMI-24 is of industrial grade. Its method of analysis and spectral data are recorded.



CONTENTS

FIGURES

TABLES

1. INTRODUCTION

1.1 Static Charge Problem

Static charge is a familiar problem. It may cause fire or explosion hazard and create processing problems in manufacturing plants especially in the textile and plastic industries. For the military, the problem is the prefiring of electronic fuzes due to static charge buildup on plastic nose cones. In artillery fuzes M514A1E1, M728, and M732, the plastic is Noryl, a modified polyphenylene oxide of General Electric Co. Noryl is a moldable thermoplastic with attractive thermal and mechanical properties. An antistatic protection is needed for the nose cone during the 20-s flight time while the peak temperature of the cone surface may reach up to 370 C (700 F) in an environment wet or dry beyond control.

1.2 Early Development of HDL Antistatic Coating Formulation

The amount of electricity involved in a static charge is small, but the voltage created is usually as high as 3 to 13 kV. It has been well established that the problem of static charge diminishes as the resistivity decreases. To increase the conductance, one can increase the moisture content of the surrounding atmosphere as is commonly done at home and also by some industries. An alternative method is to use antistatic agents applied either on the surface or by internal incorporation in the plastic. However, nearly all of the agents known are hygroscopic or water soluble. Their effectiveness depends basically on atmospheric moisture, and their durability is highly questionable during long storage.

A different approach is to use conductive carbon black, which is inexpensive and durable. Carbon black is compounded internally in automobile tires for both reinforcement and antistatic protection. Resistors of different resistivities are made with ink formulations composed of carbon black and polymer binders. To solve the static charge problem of the artillery fuzes, the Harry Diamond Laboratories (HDL) conducted an investigation from 1970 to 1971. It was found then that internal incorporation[1] of carbon black into Noryl was impractical because the level of carbon needed was so high that the moldability and the physical properties of the plastic were seriously compromised. Effort was then concentrated on the development of a carbon-containing coating formulation with a polymer binder that should crosslink on the cone surface to give a durable film. A satisfactory

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[1] *E. J. Volko and G. C. Tesoro, Encyclopedia of Polymer Science and Technology, II, John Wiley and Sons, Inc., New York (1970), 204-229.*

coating formulation for this application should possess the following desirable properties:

Convenient pot life
Rapid gelation at room temperature
Relatively low cure temperature
Good adhesion to Noryl
High thermal stability
Some flexibility
Satisfactory abrasion resistance

Among the polymers commonly used for coating formulations, epoxy resin was selected as the most suitable candidate to meet the above requirements. An antistatic coating formulation was then developed and put in use immediately for fuze M514A1E1. Figure 1 shows formulation* HDL900-3, which was developed.

Epon 1001	100.00
EMI-24	9.9
Beetle 216-8	5.1
Carbon Vulcan XC-72R	3.3
Xylene	57.8
Methyl isobutyl ketone	40.8
n-Butyl alcohol	17.3
Cure cycle:	130 ± 2 C for 2 hr or flash cure with infrared lamps

Figure 1. Antistatic coating formulation HDL900-3 (parts by weight).

Solid epoxy Epon 1001 was preferred in the formulation to the standard workhorse, liquid Epon 828, because Epon 1001 is more flexible, cures faster, and hence has shorter set-to-touch time. 2-Ethyl-4-methylimidazole (EMI-24) is a unique amine for epoxy curing

The formulation is based on HDL engineering drawing No. 11718295, revision C (17 December 1975). The original HDL formulation contains 0.30 phr of defoamer PC-1344 of Monsanto Co. The contributors to the coating development were E. L. Anderson, J. M. Boyd, R. High, E. Horsey, S. Y. Lee, J. Schmid, and G. B. Wood.

known to give properties in the middle of the range between those of aliphatic and aromatic amines. Aliphatic amines cure quickly even at room temperature, but give only low thermal stability, whereas aromatic amines give high thermal stability, but need to be cured at high temperatures that may degrade Noryl. Above all, the imidazole structure is noted to have inherently good antistatic properties.[2] Beetle 216-8 is a proprietary urea-formaldehyde solution that works as a flow control agent.

For the preparation of the coating compound, conductive carbon Vulcan XC-72R of Cabot Corp. was dispersed in the compound by milling. It should be recognized that sufficient milling is necessary to assure satisfactory conductance, but overmilling lowers conductance. The coating was applied with either a one-component or a two-component spraying gun. The sprayed coating was immediately cured in an oven at 130 C. Any delay could lower the conductance. For the stockpiled fuzes, a flash cure with infrared lamps was necessary to effect a short, but intensive, cure without overheating the electronics inside the fuze. The coating was shown to be satisfactory by many fuze tests including thermal spinning, thermal shock, vacuum steam pressure, and air gun. Figure 2 shows two nose cones coated with formulation HDL900-3.

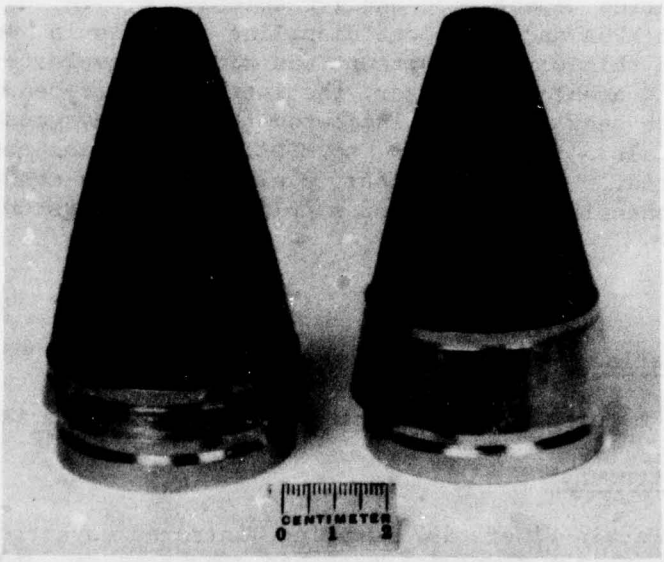

Figure 2. Coated nose cones of artillery fuzes.

[2] *V. E. Shashoua, J. Polym. Sci., Part A, 1 (1963), 169.*

1.3 Approaches to Develop Alternative Formulation

When it was realized that the supply of the curing agent EMI-24 used in HDL900-3 was from a single domestic source and that this source was not reliable at times, a program was initiated to develop an alternative coating formulation (to be called HDL900-6) using some curing agents other than EMI-24. To minimize changes of the coating process, attention was focused on the known aromatic amine curatives. (Imidazole is an aromatic heterocyclic amine.) It was recognized that no other aromatic amines could cure epoxy at temperatures as low as EMI-24 did and yield an equivalent heat deflection temperature (HDT). m-Phenylenediamine (MPDA) is one of the most popular aromatic amines and is known to give a high HDT when a full cure is obtained at 150 C or higher. However, curing at a temperature as low as 130 C is possible when 0.5 to 1.0 part of salicylic acid per hundred parts of resin (phr) by weight is used as an activator.[3,*] Therefore, MPDA was considered the prime candidate for replacing EMI-24 in the antistatic formulation.

Although MPDA can exist as a metastable liquid at room temperature, it tends to crystallize on standing. Hence, it is not that convenient to use. To circumvent the difficulty, MPDA can be mixed with another low melting amine curative to form a eutectic liquid mixture. Curing agent Z of Shell Chemical Co. is such a mixture consisting of MPDA and methylene dianiline (MDA) in a weight ratio of 60 to 40. In this work, an attempt was made to evaluate both MPDA and Shell's curing agent Z. However, the latter was dropped at the early stage because new findings indicated that MDA may cause hepatitis in some individuals. In addition to MPDA, a commercial coating material was considered. Some preliminary evaluation was conducted on the material, a phenolics formulation supplied by the Gayston Corp.

2. EXPERIMENTAL METHODS

2.1 Materials

The materials used in this work are listed in table I (p. 9).

2.2 Instruments

A Perkin-Elmer 467 grating infrared spectrophotometer, a Varian A-60 nuclear magnetic resonance spectrometer, and a Varian 1520 Aerograph (gas chromatograph) were used for material identifications

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[3] *H. Lee and K. Neville, Handbook of Epoxy Resins, McGraw-Hill Book Co., New York (1967), 4-18, 8-3.*

**Jack Klarquest, Shell Chemical Co., private communication.*

TABLE I. MATERIALS

Name	Manufacturer	Description
Epon 1001	Shell Chemical Co.	Solid epoxy resin of bisphenol A type, epoxy equivalent wt 450 to 550
Epon 1001-X-75	Shell Chemical Co.	75 percent solution by wt of Epon 1001 in xylene
EMI-24	Fike Chemicals, Inc.	2-Ethyl-4-methylimidazole for epoxy curing use, dark liquid, 80 to 90 percent pure
EMI-24	Aldrich Chemical Co.	97 percent pure
MPDA	Shell Chemical Co.	m-Phenylenediamine, off-white flakes, mp 63 to 65 C
Curing agent Z	Shell Chemical Co.	Liquid composed of MPDA and methylene dianiline in ratio of 60:40 by wt
Salicylic acid	J. T. Baker Chemical Co.	Reagent grade
Beetle 216-8	American Cyanamid Co.	Urea-formaldehyde resin used as flow control agent, 74 percent solids
Vulcan XC-72R	Cabot Corp.	Highly conductive carbon, particle size 29 nm av
Defoamer PC-1344	Monsanto Co.	Acrylic copolymer used to assist release of bubbles
PPO GFP-3-303	General Electric Co.	Polyphenylene oxide, 30 percent glass filled
Noryl GFN-2	General Electric Co.	Modified PPO, 20 percent glass filled

and analyses. An Autolab System IV computing integrator was connected to the Aerograph for data reduction. For thermal analysis, a DuPont 990 thermal analyzer was used with differential scanning calorimetry (DCS) and thermogravimetric analysis (TGA) modules.

2.3 Preparation of Carbon-Filled Coating Compounds

A 70-g batch of coating formulation HDL900-3 or HDL900-6 was made by weighing first conductive carbon and then all the rest of the ingredients except EMI-24 or MPDA in a glass jar of 5 cm o.d. × 8.5 cm height. After these materials were preliminarily mixed with a spatula, five porcelain balls of 2-cm (3/4-in.) diameter were added to the

mixture, and the jar was tightly closed with a Teflon-lined cap. The mixture was then ball milled on a paint roller at 80 rpm for 18 to 20 hr. After the balls were removed and EMI-24 or MPDA was added and hand mixed into the dispersion, the compound was ready for spray coating.

2.4 Spray Coating of Noryl Nose Cones

The jars chosen for mixing the compounds just fit Crown Industrial Products Co. 13-oz (369-g) No. 8011 Spra-Tool sprayer (pressured propellants in a can). After the jar was screwed onto the sprayer and shaken a few times, the spray was tested several times to draw the dispersion into the nozzle of the sprayer (fig. 3).

Figure 3. Spray coating of nose cone.

The Noryl nose cone of fuze M728 to be coated was cleaned by wiping with paper towels soaked with pentane and with methanol. Then the nose cone was placed on a revolving platform whose speed was set at 120 rpm by a powerstat. The spraying was done in a well-ventilated fume hood with the sprayer held about 18 cm (7 in.) from the cone and the coating applied from the cone bottom up. The coating was cured without delay.

2.5 Electrical Resistance Measurement

2.5.1 Volume Resistivity

Molded PPO plaques (GFP-3-303) of 5 × 5 × 0.3 cm, were used as coating substrates for resistivity measurements. The thicknesses were accurately measured with a micrometer, and the plaques were cleaned by wiping with paper towels soaked with pentane and with methanol before spray coating. After the coating was cured and cooled, an approximately 1-cm-wide strip of conductive silver paint (DuPont Co. conductive paint No. 4817) was hand painted over the coating on each of two opposite ends. The edges of the plaques were scraped with a razor to remove all coating materials so that the coated surface to be measured was isolated electrically. Resistance was measured with either a Keithley Co. megohmmeter model 510 or a Sencore Co. field effect meter model FE-16. The megohmmeter covers the resistance range of 10 to 10^7 megohms and has a built-in calibration mechanism, whereas the field effect meter measures 1 to 500 megohms and was calibrated with an Eico Co. model 1100 resistance box. The volume resistivity, ρ, was calculated from the measured resistance, R, according to the following equation as described[4] in ASTM 257-66:

$$\rho = R\left(\frac{tw}{\ell}\right) ,$$

where

t = thickness of coating (centimeters),

w = width of coated surface or length of silver paint strip (centimeters),

ℓ = length of coating surface or distance between two silver paint strips (centimeters).

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[4] *Tests for D-C Resistance or Conductance of Insulating Materials, American Society for Testing and Materials, Philadelphia, ASTM 257-66 (1966).*

2.5.2 Nose Cone Resistance

The electrical resistance of the nose cone was measured with the field effect meter. The cone was held in a fixture that has an upper and a lower contact ring. The gold-plated contact rings fit the top or bottom part of the cone as shown in figure 4.

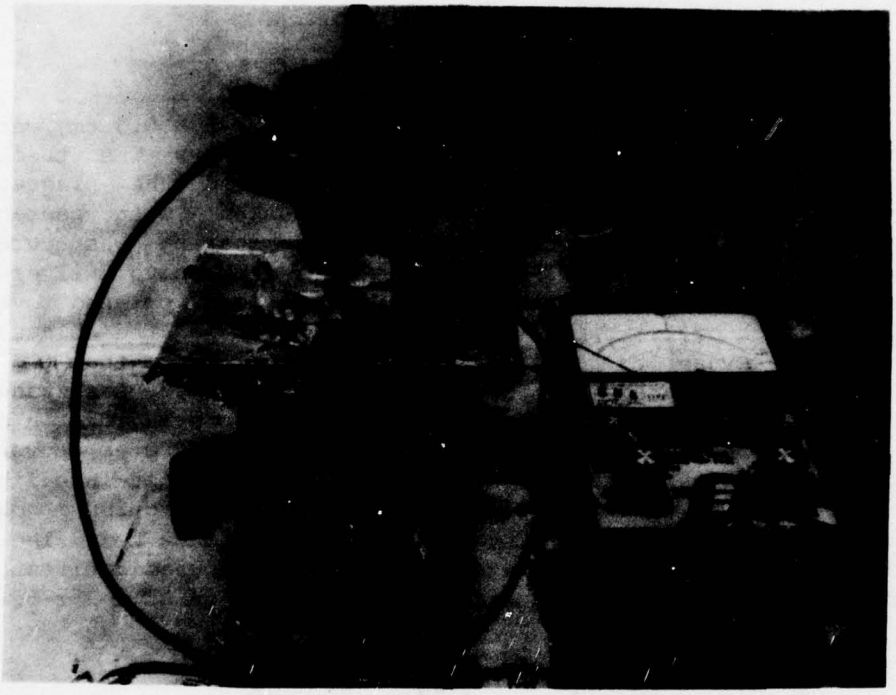

Figure 4. Measurement of resistance of nose cone.

3. RESULTS AND DISCUSSION

3.1 Curing Agent Level and Curing Conditions

When MPDA is used to cure epoxy, it is customary to adopt a two-stage cure cycle to avoid the excessive exotherm that would occur if a one-stage high temperature cure were applied. A common cure

cycle[3,5] is 85 C for 2 hr and then 150 C for 2 hr. With salicylic acid used as an activator, a cure at 80 C for 1 hr and then 140 C for 1 hr was initially adopted to study the effect of curing agent levels. The stoichiometric amount of MPDA to cure Epon 1001 is 5.4 phr. Compounds M1, M2, and M3, formulated with 5.0, 6.0, and 7.0 phr of MPDA, were made as shown in table II. A control compound was formulated with EMI-24. Figure 5 compares their thermal stabilities by TGA. Apparently, 6.0 phr of MPDA is an optimal level as evidenced by M2, whose thermal stability appears to be slightly superior to that of the control.

TABLE II. FORMULATIONS FOR MPDA LEVEL STUDY

Ingredients	Formulation (parts by weight)			
	M1	M2	M3	Control
Epon 1001	100.0	100.0	100.0	100.0
MPDA	5.0	6.0	7.0	-
Salicylic acid	0.5	0.5	0.5	-
Solvent mix*	115.8	115.8	115.8	115.8
EMI-24	-	-	-	9.9
Cure cycle†	80 C for 1 hr + 140 C for 1 hr			130 C for 2 hr

*The solvent mix was made of xylene, methyl isobutyl ketone, and n-butyl alcohol in the ratio of 57.7, 40.8, and 17.3 by weight.

†For M1, M2, and M3, it was an initial cure cycle without optimization. For the control, it was the cure cycle specified for the coating formulation HDL900-3.

The good thermal stability of M2 was very encouraging. It led to the belief that, by curing with MPDA without the use of activator salicylic acid, a coating comparable to the existing coating HDL900-3 could be made. This meant that an alternative formulation could be made by replacing EMI-24 with MPDA at a chemical equivalent level. Therefore, in the curing condition study that followed, salicylic acid was eliminated and Beetle 216-8 was added in formulation M2a as shown in table III. To find an optimal cure cycle for M2a, the cure times at 80 and 140C were varied as shown in table IV. Their residual cure exotherms as recorded in DSC thermograms were compared. The residual cure exotherm measurement is simple and convenient to show the

[3] *H. Lee and K. Neville, Handbook of Epoxy Resins, McGraw-Hill Book Co., New York (1967), 4-18, 8-3.*

[5] *Epon Resins for Casting, Shell Chemical Co. (1967), 77.*

comparative degrees of cure achieved by different cure cycles. A cure at 140 C for 15 min alone left a residual exotherm barely measurable by DSC. A cure at 80 C for 30 min left a residual exotherm about half cure. A combination of 80 C for 15 min and then 140 C for 15 min left essentially no residual exotherm measurable by DSC.

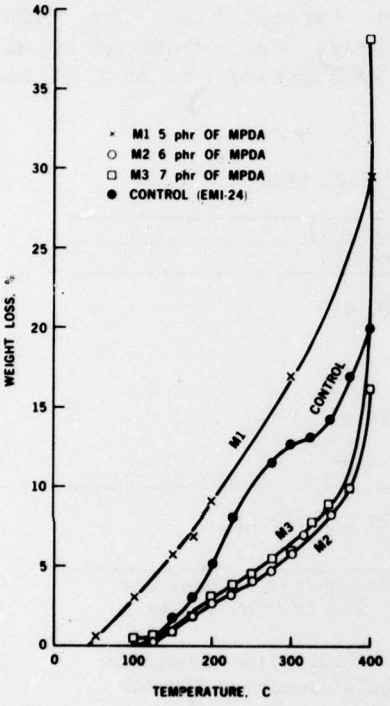

Figure 5. Thermal stabilities of compounds measured by thermogravimetric analysis.

TABLE III. FORMULATIONS FOR CURING CONDITION STUDY

Ingredients	Formulation (parts by weight)	
	M2a	Control A
Epon 1001	100.1	100.0
MPDA	6.0	-
EMI-24	-	9.9
Beetle 216-8	5.1	5.1
Solvent mix	115.5	115.8
Cure cycle	Varied	130 C for 2 hr

TABLE IV. CURING CONDITION STUDY OF FORMULATION M2a MEASURED BY DIFFERENTIAL SCANNING CALORIMETRY

Cure time (min) 80 C + 140 C		Relative residual cure exotherm	Remarks
0	0	100	Room temp mix as control
0	5	19	
0	10	1	Slightly tacky
0	15	1	
15	0	67	
30	0	55	
45	0	35	
60	0	2	
15	15	0	
30	15	0	
45	15	0	

Note: Differential scanning calorimetry samples were prepared by laying films of 0.13 to 0.25 mm (5 to 10 mils) thick with the aid of some solvent, which was later removed before the thermal analysis.

Further study was conducted by the more sensitive TGA. Table V records the thermal stabilities of M2a measured by TGA of compounds cured by varied cure cycles. The cure time at 140 C was varied from 15 to 60 min with a fixed initial cure at 80 C for 30 min. The data of weight loss temperature show clearly that a 30-min postcure at 140 C gave significantly better thermal stability than the 15-min postcure, and a longer postcure even gave an adverse effect. Therefore, the cure cycle of 80 C for 30 min plus 140 C for 30 min appears to be the best and gave a thermal stability somewhat better than that of the control.

TABLE V. THERMAL STABILITIES OF FORMULATION M2a MEASURED BY THERMOGRAVIMETRIC ANALYSIS

Formulation	Cure cycle	Temp (C) when weight loss occurred			Remarks
		Onset	1% loss	5% loss	
M2a	80 C for 30 min + 140 C for 15 min	57	109	176	Least stable
	30 min	112	210	>250	Most stable
	45 min	110	159	228	
	60 min	105	154	228	
Control A	130 C for 120 min	60	150	209	

Note: Heating rate was 10 C/min.

3.2 Coating Formulations and Their Properties

Conductive carbon was incorporated into M2a to form antistatic coating formulation HDL900-6 as shown in figure 6. The new formulation is comparable to HDL900-3. The main difference is the curing agent, 6.0 phr of solid MPDA being used instead of 9.9 phr of liquid EMI-24. Epon 1001 solution (75 percent by weight in xylene) is prescribed in the formulation for practical convenience. The resultant increase in xylene and change of the three-solvent ratio did not appear to affect the coating process. However, the amount of xylene may be easily adjusted to keep the same solvent ratio as in HDL900-3 if it is found advantageous in production.

Epon 1001-X-75	133.3 (equivalent to 100.0 of Epon 1001)
MPDA	6.0
Beetle 216-8	5.1
Carbon Vulcan XC-72R	3.3
Xylene	57.8
Methyl isobutyl ketone	40.8
n-Butyl alcohol	17.3

Cure cycle: 80 C for 30 min plus 140 C for 30 min

Figure 6. Antistatic coating formulation HDL900-6 (parts by weight).

The coating compound was made by first ball milling carbon in the binder solution without MPDA. The MPDA was then mixed into the carbon dispersion before the coating operation. Rectangular plastic plaques were spray coated with HDL900-6, and some were coated with HDL900-3 for comparison. Volume resistivities of the coatings were determined according to ASTM 257-66. Noryl nose cones of artillery fuze M728 also were coated. The coating resistivities and the coated cone resistances obtained are recorded in tables VI and VII. The data indicate that the resistivity of HDL900-6 falls in the antistatic range required and that both HDL900-6 and HDL900-3 impart similar resistances to the nose cones.

The thermal stabilities of the two coatings were compared by TGA (fig. 7). The two coatings are about the same in terms of thermal stability. The coating adhesion to the substrate Noryl is usually not a concern because epoxy bonds well to Noryl when the epoxy is cured.

In production, the degree of cure of the coating is checked by wiping the coating with a methyl-ethyl-ketone saturated cotton swab on a toothpick. A black coloration of the cotton indicates an incomplete cure. The simple tape adhesion method described[6] in ASTM D3002-71 can be used to make a qualitative check on adhesion. Both the HDL900-6 and HDL900-3 coatings showed no stripping when Scotch tape No. 105 was pressed on and removed from coatings that had been cut with a razor (parallel cut method).

TABLE VI. RESISTIVITIES OF ANTISTATIC COATINGS

Coating	Plaque (No.)	Coating thickness (mil)	Volume resistivity (ohm-cm)
HDL900-6	15	5.5	5.5×10^3
	16	5.5	4.4×10^3
	17	6.1	4.6×10^3
	18	6.4	3.3×10^3
	19	6.3	5.1×10^3
HDL900-3	1	2.6	7.0×10^4
	4	2.5	4.0×10^4
	7704	4.3	1.4×10^4
	7705	4.3	2.6×10^4
	7706	3.5	2.2×10^4

TABLE VII. RESISTANCES OF COATED NOSE CONES

Coating	Cone (No.)	Resistance (megohm)
HDL900-6	1	2.2
	2	2.9
	3	0.8
	4	0.8
HDL900-3	1	0.5
	2	0.9
	3	9.0

[6] *Standard Recommended Practice for Evaluation of Coatings for Plastics, American Society for Testing and Materials, Philadelphia, ASTM D3002-71 (1971).*

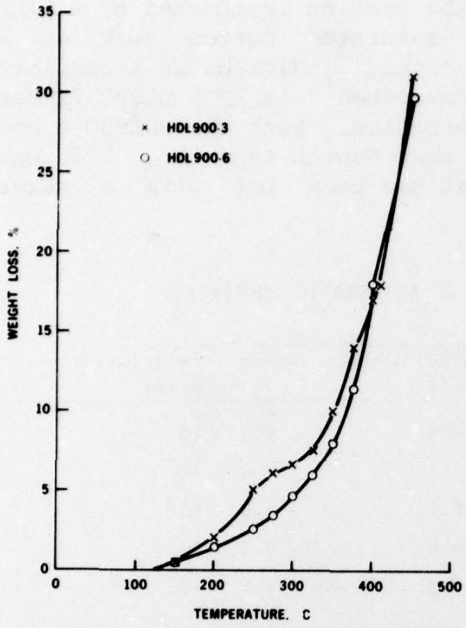

Figure 7. Thermal stabilities of antistatic formulations measured by thermogravimetric analysis (heating rate = 10 C/min).

3.3 Analysis of EMI-24 and MPDA

Two sources of EMI-24 were found available for industrial uses. An EMI-24 of 99-percent purity is imported from West Germany and distributed by BASF Wyandotte Corp. The other EMI-24 is of 80- to 90-percent purity made by Fike Chemicals, Inc., at Nitro, VA. The latter one has been used for the antistatic coating formulations and by epoxy manufacturers or formulators such as Shell Chemical Co. and Hysol Corp.

For epoxy curing, our experiments showed that the Fike EMI-24 worked as effectively as the 97-percent-pure EMI-24 from Aldrich Chemical Co. when they were used in equivalent amounts. The cured properties produced by the two EMI-24's were essentially equal. For the quality control, the infrared and nuclear magnetic resonance (NMR) spectra of the 80- to 90-percent-pure EMI-24 and the 97-percentpure EMI-24 were recorded (app A).

To conduct gas chromatographic analysis, columns made of Porapack R, Carbowax 20M, Silicone SE-30, and Silcone OV-101 were tried without success. Only the column used by Fike was found to give satisfactory separation. The column is made of 11.5-percent polyphenyl

ether (five rings) and 11.5-percent Carbowax 20M. A chromatogram of our analysis of EMI-24 with the column is shown in the appendix. The results of the analysis of two lot samples are recorded in table A-I. The EMI-24 peak was confirmed with the sample of 97-percent purity. The purity of the two lot samples ranges from 78 to 85 percent. Some variations in the computed percentages occurred between runs of the same sample. They are related only to the very small overlapping peaks, which were neglected sometimes by the computing integrator. Nevertheless, the gas chromatography method appears to be the best method for the quality control of EMI-24.

The MPDA used was quite pure by the melting point determination. Under the same gas chromatography condition, the MPDA showed only one peak, with a retention time of 1986 s (33.1 min).

3.4 Gayston's Coating Material

Gayston's conductive coating material was evaluated as a possible replacement for HDL900-3. It is a formulation of phenolic resin filled with conductive carbon and other fillers up to 40 to 50 percent of the total solids. It is made of two components: low resistance GCA53776 and high resistance GCB34577. According to Gayston, when they are mixed in a ratio of 5 to 95 by weight to make a coating material that is cured at 121 C for 1 hr, the coating resistance should be good for antistatic use.

Samples of the material and the coated nose cones were received for evaluation. The thermal stability of the cured material was found by thermal analysis to be excellent. However, its adhesion on Noryl was very poor in comparison with HDL900-3. The resistance of the coated cone also was higher than that required for antistatic protection. Although the supplier might be able to make improvements, the evaluation was not pursued further because it was not considered urgent to find a replacement completely different from HDL900-3.

4. CONCLUSIONS

An alternative antistatic coating formulation, HDL900-6, was developed with m-phenylenediamine used as the curing agent. The new formulation may be used in place of the earlier HDL900-3 when the supply of the latter's curing agent EMI-24 runs short. The replacement was designed to ensure minimum changes in the coating process. Both coatings are conductive carbon filled epoxy. Their thermal stabilities are essentially the same. The EMI-24 used is of industrial grade, 78 to 90 percent pure. The method of analysis and its infrared and NMR spectra are documented in appendix A for reference in this report.

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(4) Tests for D-C Resistance or Conductance of Insulating Materials, American Society for Testing and Materials, Philadelphia, ASTM 257-66 (1966).

(5) Epon Resins for Casting, Shell Chemical Co. (1967), 77.

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APPENDIX A.--SPECTRA AND ANALYSIS OF MATERIALS

APPENDIX A

Figures A-1 to A-6 show the infrared and NMR spectra and the chromotogram of EMI-24 and Beetle 216-8. Table A-1 lists gas chromatographic analysis data.

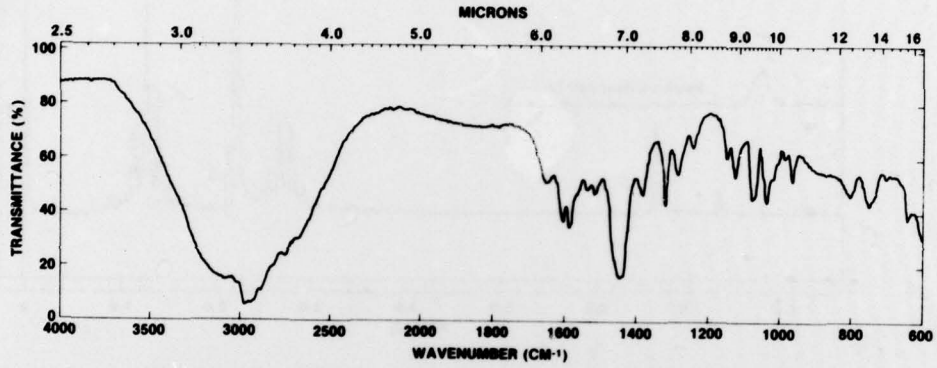


Figure A-1. Infrared spectrum of EMI-24 labeled 80 to 90 percent pure (sample smeared on NaCl plates).

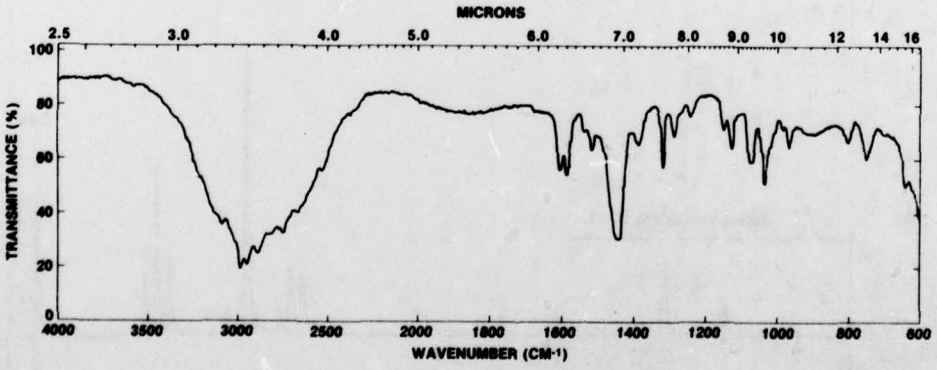


Figure A-2. Infrared spectrum of EMI-24 labeled 97 percent pure (sample smeared on NaCl plates).

APPENDIX A

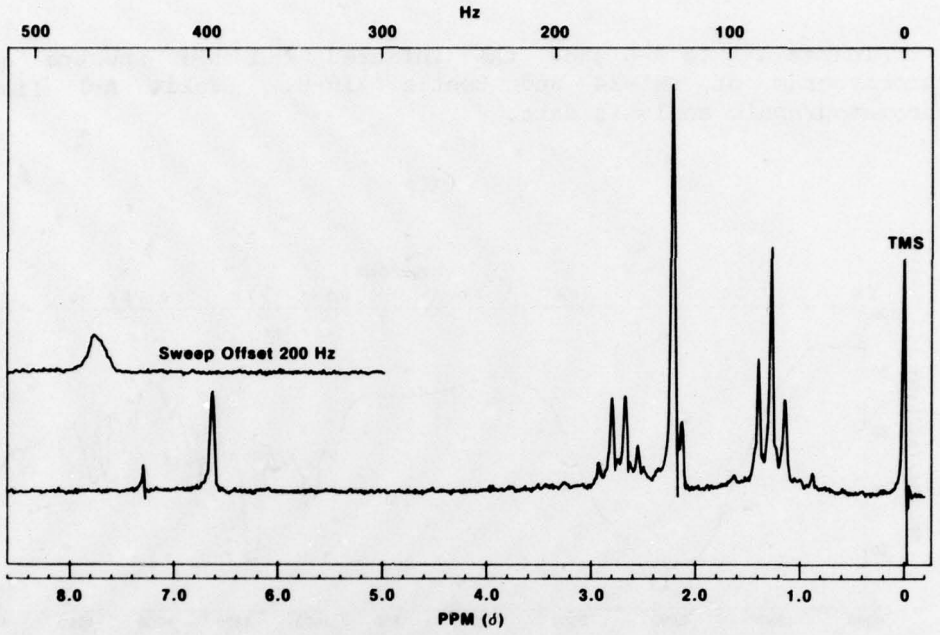


Figure A-3. NMR spectrum (60 MHz, $CDCl_3$) of EMI-24 labeled 80 to 90 percent pure.

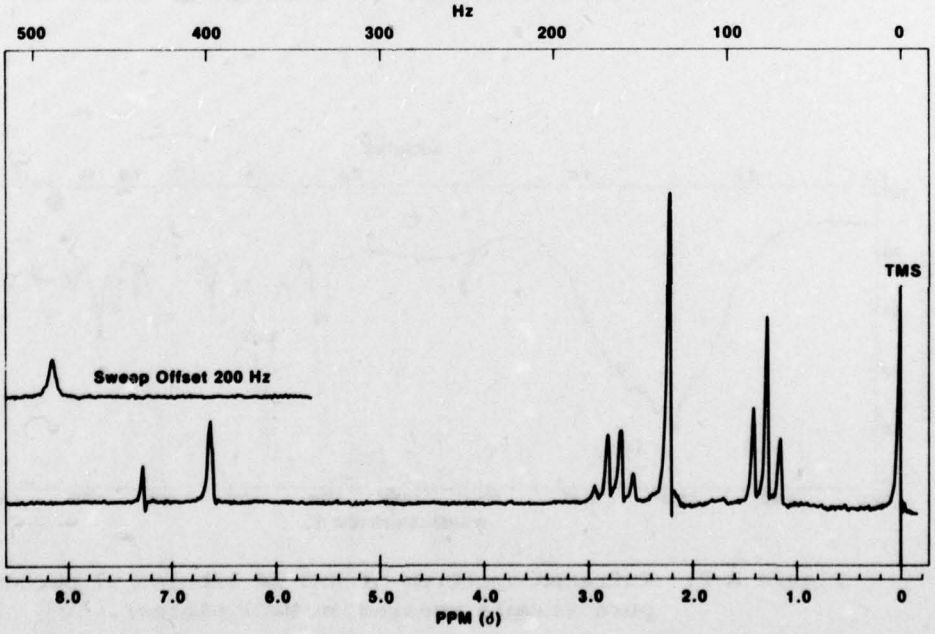


Figure A-4. NMR spectrum (60 MHz, $CDCl_3$) of EMI-24 labeled 97 percent pure.

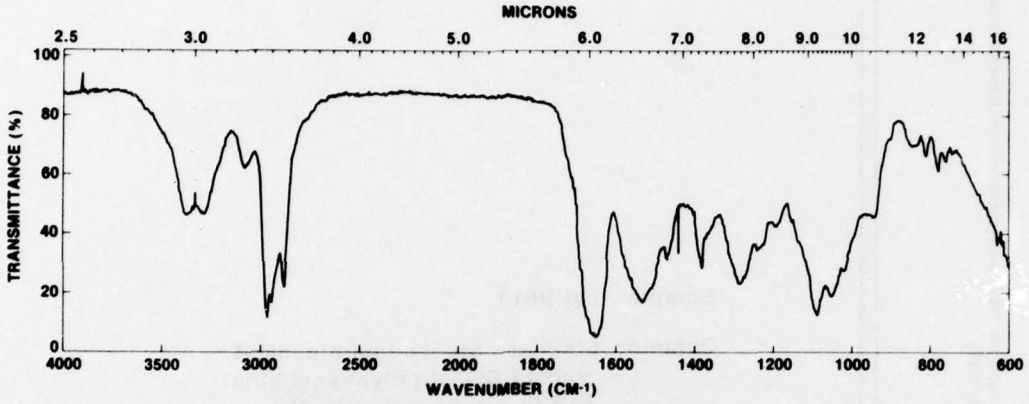
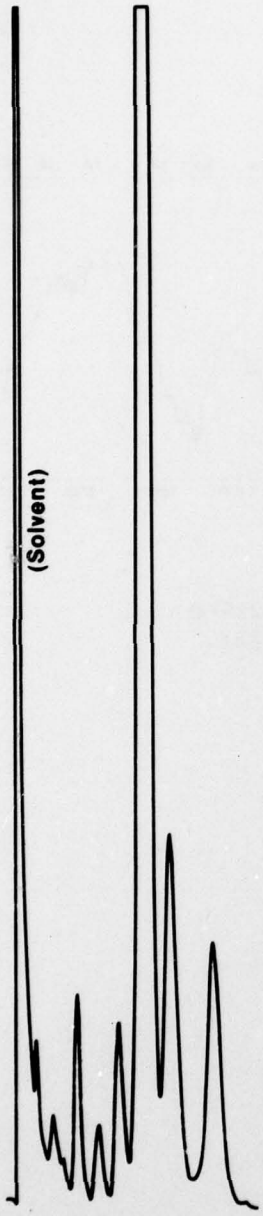


Figure A-5. Infrared spectrum of Beetle 216-8 (sample smeared on NaCl plates).

APPENDIX A



Sample: Lot B613

Column: Stainless Steel column packed with 11.5% of polyphenyl ether and 11.5% Carbowax 20M on Chromosorb G-NAW, 60-80 mesh

Column Temp: 220 C

Chromatogram recording: in log scale

Figure A-6. Gas chromatographic analysis of EMI-24 (Fike Chemicals, Inc.).

APPENDIX A

TABLE A-1. GAS CHROMATOGRAPHIC ANALYSIS* OF EMI-24

Lot B613, received 1972†				Lot F1098, received 1976†			
Run 1		Run 2		Run 1		Run 2	
Retention (s)	Purity (%)	Retention (s)	Purity (%)	Retention (s)	Purity (%)	Retention (s)	Purity (%)
112	0.28	111	0.26	-	-	-	-
138	0.02	-	-	-	-	-	-
193	0.55	193	0.70	193	1.99	192	1.89
-	-	236	0.07	-	-	-	-
310	2.58	310	2.51	309	0.40	308	0.41
411	1.27	412	1.05	412	1.63	410	1.72
513	3.51	513	3.38	-	-	510	0.61
645	78.05	646	78.51	645	84.99	645	84.16
762	6.88	764	6.72	763	1.89	761	1.92
973	6.85	975	6.78	972	9.11	970	9.30

*Stainless steel column, 3.2 mm × 2.1 m, packed with 11.5 percent of polyphenyl ether (five rings) and 11.5 percent Carbowax 20M on Chromosorb G-NAW, 60 to 80 mesh; column temperature, 220 C; injector temperature, 284 C; helium as carrier gas at flow rate of 32 ml/min.

†Lots from Fike Chemicals, Inc.

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